RESL TECHNICAL PROCEDURE

CHEM-TP-GA/B.2

GROSS ALPHA AND BETA IN WATER AND WASTE WATER

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TITLE: CHEM-TP- GA/B.2, GROSS ALPHA AND BETA IN WATER AND WASTE WATER

PURPOSE

The purpose of this procedure is to describe the method used to determine gross alpha and beta activity in water and waste water.

APPLICABILITY

This procedure is applicable to water and waste water samples containing unknown quantities of dissolved solids. This procedure is not applicable to samples prepared for counting outside RESL.

RESPONSIBILITIES

RESL staff responsible for implementing this procedure are:
Radiochemist(s)

DEFINITIONS

Covered hotplate - A hotplate covered with a fiberglass mat.

PROCEDURE

1 **ABSTRACT** Gross alpha and beta measurements are mainly used for screening purposes. Gross measurements are a shorter and cheaper method than determination of specific nuclides. Depending upon the purpose for the analyses, gross alpha or gross beta may suffice. Specific analyses may be requested if the alpha activity is greater than 5 pCi/L and/or the gross beta activity is greater than 8 pCi/L. Samples are evaporated with excess nitric acid and transferred to tared stainless steel plates or serrated planchets. A counting efficiency based on mg of absorber over the range 0-160 to 0-300 for alpha and beta, respectively, is used for each sample. The reference nuclide for alpha activity is ^{239}Pu and for beta activity is ^{137}Cs.

2 LIMITATIONS AND INTERFERENCES The nitrate salts can be hygroscopic so it is necessary to store the prepared plates in a desiccator prior to counting to minimize the effect on counting efficiency (C.E.). The C.E. for the gross alpha varies because the salts are not distributed evenly on the plate. Since it is nearly impossible to distribute the salts evenly, there is a large uncertainty associated with gross alpha results.

3 Safety Precautions

3.1 Wear safety glasses, a face shield (optional), a laboratory coat, and nitrile gloves to protect the eyes and skin from both chemical and radiological hazards. Use caution in handling HNO_3 and avoid spills.

3.2 Use caution to avoid burns while using the hotplate or heat lamp.

3.3 Frisk hands, coat, and work areas when work is completed using radioactive material as defined in Step 6.2 below.

4 EQUIPMENT NEEDED

4.1 Fume hood

4.2 Hotplate, 3600 W, 46 x 61 cm covered with a 1.6-mm thick fiberglass mat

4.3 Heat lamp(s), 250 W

4.4 Plates, 4.76-cm diameter by 0.06-cm thick, flat, type 304 stainless steel

4.5 Planchets, 5.08-cm diameter, serrated, type 304 stainless steel with a 0.32-cm wall

4.6 Pipets for use with disposable tips, 1 mL and adjustable from 1 mL to 10 mL

4.7 Beakers, glass, 50, 100, 150, 250 mL

4.8 Beaker tongs

4.9 Desiccator, to hold prepared samples

4.10 Gross alpha and beta counters (See CHEM-TP-CA.02)

5 **CLEANING PLATES AND PLANCHETS** Remove the sticky paper from the polished side of each flat plate and rub the surface of the plate with a tissue moistened with acetone to remove residual adhesive. Rinse the plates with demineralized water. Clean a supply of plates and planchets by immersing them in 8 M HNO_3 in a beaker of appropriate size. Cover the beaker with a watch glass and heat until the acid has boiled for at least 15 min. Cool, rinse the plates and planchets with demineralized water, air dry them, and store them in a covered container for use as needed.

6 SAMPLE PREPARATION

6.1 Verify that when a water sample is received, it is 2% v/v in concentrated HNO_3. Samples containing paper pulp must be reconstituted as described in Procedure CHEM-TP-SP.02. If the sample has not been acidified when received, make the sample 2% v/v with concentrated HCl (or HNO_3 if HNO_3 will not interfere with other requested analyses) and allow it to stand overnight before analysis.

6.2 Determine the quantity of a sample to be analyzed from several factors. These factors are the total amount of sample received, the amount of sample used for other analyses requested, the amount of sediment or dissolved solids in the sample, and the gross gamma count. If the quantity of sediment is "small", the sample should be shaken vigorously and aliquanted in an attempt to obtain a representative portion of solids with the liquid. If, in the analyst's judgment, too much sediment is present, the

aliquant should be obtained by careful decantation after the sample has stood overnight. The volume used from lagoon, settling pond, or other samples likely to contain large quantities of dissolved solids must be limited. When total sample, sediment, or dissolved solids are not limiting factors, the following table may be used as a guide for gross beta aliquants on in-plant liquid wastes from nuclear power plants. The largest possible volume and longest counting times (1000 min for alpha and 60 min or more for beta) should be used when sensitivities of a few pCi/L are required as is generally the case with potable or other clean natural waters.

Gross Gamma Count[1]	Volume for Beta
$<10^4$ c/m	100 mL
10^4 to 10^5 c/m	50 mL
10^5 to 5 x 10^5 c/m	5 mL
$>5 \times 10^5$ c/m	1 mL

NOTE: ANY .. EMITTER WITH ≥5000 dpm OR ANY ALPHA EMITTER WITH ≥500 dpm (ACCOUNTING FOR DETECTOR EFFICIENCY) MUST BE LABELED AND CONTROLLED AS RADIOACTIVE MATERIAL.

6.3 **Samples Acidified with HNO_3 - Direct Pipetting** Tare plates and planchets to the nearest 0.1 mg and record the weight(s). Place the plate or planchet on an inverted 50-mL beaker under a heat lamp. Make the plate or planchet reasonably level. Pipet up to 5 mL of sample for gross alpha directly onto the plate or up to 10 mL of sample for gross beta onto the planchet. If the liquid does not completely cover the surface of the plate or planchet, add 4 M HNO_3 until the surface is covered. Continue with Step 6.7. Begin with Step 6.4 for samples larger than 5 mL for gross alpha or 10 mL for gross beta but omit the addition of excess HNO_3.

6.4 **Large Samples and All Samples Acidified with HCl** Use a pipet or graduated cylinder to measure the sample and transfer it to a beaker of appropriate size. Rinse the graduated cylinder with a little 4 M HNO_3 and add the rinse to the beaker. Add an excess of 16 M HNO_3 to samples that were acidified with HCl. Place the beaker on a covered hotplate and evaporate the sample to about 3 mL. Rinse down the sides of the beaker with about 5 mL of 16 M HNO_3 and cover it with a watch glass. Evaporate the sample to 1 to 2 mL.

6.5 Remove the sample from the hotplate and allow it to cool.

6.6 Use 4 M HNO_3 as a rinse to transfer the sample to a tared SS plate or planchet which has been mounted on an inverted 50-mL beaker. A final rinse with 2 drops of 1:1 H_2O:HNO_3 to remove solids from the beaker may be used.

[1] See CHEM-TP-G.03

6.7 Evaporate the sample to dryness under a heat lamp at a distance of about 10 cm. Place the plate or planchet directly on a bare hotplate for 1 min or longer until fuming stops. Remove the plate or planchet from the hotplate and allow it to cool slightly. Add, to the planchet only, enough drops of a glue-acetone solution (30 drops of model airplane dope in 200 mL of acetone) to wet the solids on the planchet. This treatment is used to fix loose particles to the planchet and prevent contamination of the beta detector. It is not necessary for alpha plates.

6.8 Place either the plate or planchet in a desiccator containing anhydrous $Mg(ClO_4)_2$ and indicating Drierite until the plate or planchet reaches room temperature.

6.9 Reweigh the plate or planchet to determine the mg of absorber. Mount the alpha plate under a prebackgrounded, silver-activated zinc sulfide phosphor and place the mounted sample back in the desiccator for at least 4 hr to allow Rn daughters to decay. Place the beta planchet in a holder and count. Count time for gross alpha or beta varies depending upon the amount of sample used and/or the sensitivity required for the analysis.

7 **CALCULATIONS** Calculate the results and their uncertainties with the menu-prompted VAX RESULT computer program for gross alpha and beta. Use a yield of 98% for samples not directly placed on plates or planchets. Other data required are the gross count, the background count, mg of absorber, volume of sample in mL, and counting time in minutes. The count time must be the same for the sample and background. Use a volume corrected for dilution by the acid added to the original sample, e.g., a 100-mL aliquant of sample made 2% (v/v) with HCl represents 98 mL of original sample.

REFERENCES

CHEM-TP-CA.2 Gross Alpha and Beta Counting Efficiencies - Liquids

QUALITY RECORDS

Hard copy record of results.